

# PRV

PATENT- OCH REGISTRERINGSVERKET

Patentavdelningen

## Intyg Certificate



*Härmed intygas att bifogade kopior överensstämmer med de handlingar som ursprungligen ingivits till Patent- och registreringsverket i nedannämnda ansökan.*

*This is to certify that the annexed is a true copy of the documents as originally filed with the Patent- and Registration Office in connection with the following patent application.*

(71) Sökande                      Uponor Innovation AB, Fristad SE  
Applicant (s)

(21) Patentansökningsnummer    9503272-8  
Patent application number

(86) Ingivningsdatum                      1995-09-20  
Date of filing

Stockholm, 2005-06-16

För Patent- och registreringsverket  
For the Patent- and Registration Office

Hjärdís Segerlund

Avgift  
Fee            170:-

**BEST AVAILABLE COPY**

SÖKANDE: Uponor B.V.

UPPFINNINGENS BENÄMNING: Improved extruded hollow products

5

The invention relates to a method of extruding a product of plastic material.

10 Several methods for orientation of the material of a polyethylene pipe have been proposed but none of them has come into commercial use so far. Polyethylene which is a highly crystalline material can be successfully oriented below the crystalline melting point only by using very high  
15 stretching forces in a die-drawing batch process. Above the crystalline melting point orientation can be effected during extrusion of the pipe but only in a very narrow temperature range. A great problem in that case is that the orientation disappears rapidly and that only thin-walled  
20 products can be cooled rapidly enough for maintaining the orientation.

Considering these practical limitations of prior art the invention provides a method of the kind referred to above which according to a more general aspect has obtained  
25 the characterizing features of claim 1.

The method of the invention would be of commercial interest and is well suited for industrial application in manufacturing polyethylene pipes having a relation between wall thickness and diameter which is  $\geq 3:100$  by a  
30 continuous process, particularly due to the fact that the temperature range over which orientation of the polyethylene material can be achieved is dramatically broadened: the temperature typically ranges from 135° to 250°C.

35 In the method of the invention a crosslink agent is added to the plastic material in order to facilitate orientation of the material and thus to provide a method of

orientation which is suitable for practical commercial use. The addition of such agent is known per se in order to obtain crosslinking of the material in extrusion of pipes. However, even if expansion of the pipe has been effected in  
5 connection with the extrusion the sole purpose of adding a crosslink agent has been to obtain crosslinking. In this connection reference is made to DE-A-2 051 390 which describes a method of continuous manufacture of pipes of crosslinked polyolefines wherein the completely crosslinked  
10 material is heated, expanded and then cooled in the expanded state in order to improve the strength properties of the pipe. The expansion is effected by means of a mandrel. There is no mentioning of the degree of expansion and also no mentioning of orientation being effected by the  
15 expansion.

WO-A-8401920 describes a method for orientation of the material of plastic pipes wherein the pipe is passed through a heated jacket in order to crosslink the material of the plastic pipe and wherein the crosslinked pipe inside  
20 the jacket is expanded by internal pressure to engage the inside of a wider portion of the jacket. There is no mentioning of the addition of crosslinking agent and no disclosure of axial orientation and cooling of the plastic pipe.

25 According to a further aspect of the invention the manufacture of hollow products, typically pipes, with tailor-made properties is facilitated. By the addition of crosslink agent to only that section of the product that is to be oriented, products with greatly varying properties  
30 can be made, such as products wherein for example inside layers are made of non-oriented materials to have better abrasion resistance, while an outer layer of pigmented non-oriented material can be attractive due to better welding properties. Considering drinking water quality an  
35 especially alternative for the non-crosslinked inside

material is such plastic that is impermeable to rest products which stay in the crosslinked section due to chemical reactions during crosslinking.

The invention also relates to a hollow product, e.g. a pipe, according to claim 22, an extruder line according to claim 36, and a method of joining pipes according to claim 46.

The invention will be described in more detail with reference to the accompanying drawings in which

FIG 1 discloses a pipe extrusion line for practising the method of the invention in axial cross-sectional view, and

FIG 2 discloses in a similar view of another embodiment of the pipe extrusion line for practising the method of the invention.

In FIG 1 there is fragmentarily shown a die 10 and a fixed core which form part of an extrusion head of a conventional pipe extruder (screw extruder) and define an annular die opening. The core projects from the extrusion head and forms at the free end thereof a mandrel 11A.

It should be mentioned here that certain materials with very high molecular weight may require ram extruders (piston extruders) or the like instead of conventional screw extruders. Also multilayer products can be extruded by means of ram extruders by applying the crosshead technology. It is also very important that at least the layer to be oriented preferably is extruded with a tooling system which is totally spider free, i.e. the mandrel is supported upstream of the material flow and hence gives a flow without any weldlines. The need for this is due to brittleness of glassy state crosslinked polyolefine. Any spiders in the flow of material which has begun to crosslink will have detrimental effects to the hoop strength of the product. Very effective in minimizing the detrimental effects of weldlines are certain cross-heads

1995-09-20

Huvudfaxen Kassar

with rotating die-sets. Rotating mandrel with counter-rotating sleeve can also give desired fiber orientation in the hoop direction in case that fibers are added to the plastic material.

5 The plastic material to be extruded can be olefin (co)polymer which throughout this specification is meant to include olefin homopolymers, copolymers or melt blends of two or more (co)polymers which either inherently or as a consequence of melt blending have the desired haul-off-  
10 tension, molecular weight and molecular weight distribution characteristics.

Preferably the olefine (co)polymer to be extruded should have a density which is at least  $900 \text{ kg/m}^3$ , suitably above 920 and preferably from 930 to  $960 \text{ kg/m}^3$ .

15 The definition of polyethylene in this context includes copolymers of ethylene with at most 5 % by weight of an alkene-1 with 3 or more carbon atoms.

In the preferred embodiment as described below the material should be HD polyethylene with the addition of  
20 organic peroxides as crosslink agent for crosslinking during extrusion and phenolic antioxidants, the addition of peroxides and antioxidants being totally 0.1 - 0.7 % by weight of the polyethylene, preferably 0.25 - 0.5%.

Generally, the material to be crosslinked can be any  
25 crosslinkable extrudable material such as crosslinkable plasticized PVC.

Also silanes can be used as crosslink agent for crosslinking of material sections of the finished tubular product in water oven.

30 The material is plasticized in the extruder and is discharged from the extrusion head as a cylindrical tubular member 12 having a large wall thickness. At the discharge opening of the extrusion head there is provided a heater 10A such as a radiation heater, for heating the tubular  
35 member to a temperature which is sufficient in order to

crosslink the material thereof to a degree ranging from 10 to 100 %. Downstream of heater 10A there are provided along the path of the tubular member two opposite circulating trains of concatenated mold halves 13 which are moved in an  
5 endless path over drive sprockets 14. Along the path of the tubular member the mold halves are guided by means, not shown, to come together at mandrel 11A and to form a bipartite mold forming a cylindrical mould cavity and enclosing the tubular member the mold halves being driven  
10 along the path of the tubular member in the moving direction thereof at the same speed as that of the said member. A mandrel 15 is located inside the tubular member and is attached to the extrusion head by means of a bar 16. Through a passage in the bar a gaseous fluid such as air or  
15 inert gas is supplied to the interior of the tubular member in the space defined between mandrel 11A and mandrel 15 in order to keep the wall of the tubular member engaged with the surface of the bipartite mold cavities. The mold halves 13 are heated at a suitable location in the endless  
20 circulation path thereof, e.g. at 17, by suitable heating means operating with fuel burners or electric resistance elements. When the pipe wall contacts the heated bipartite molds heat will be supplied to the polyethylene material to maintain said material at the crosslinking temperature  
25 during a period which is sufficient in order to reach the desired degree of crosslinking.

Downstream of mandrel 15 a plug 18, preferably of balloon type, is provided in the pipe said plug being anchored to mandrel 15 by means of a rod 19. Pressurized  
30 fluid is supplied to the balloon plug through passages in rods 16 and 19 to keep the plug inflated in sealing engagement with the inside surface of the tubular member. In the space between mandrel 15 and plug 18 a pressure is maintained by means of fluid such as air or inert gas  
35 supplied to said space through passages in rods 16 and 19

1995-09-20

Huvudfaxen Kassar

said pressure being higher than the pressure maintained in the tubular member between mandrel 11A and mandrel 15. The tubular member which is still soft will be exposed to free expansion radially for hoop stretching of the wall thereof under the influence of this higher pressure to a pipe with a larger diameter than that of the tubular member leaving the extruder, and with a wall thickness that is reduced in relation to the wall thickness of said member. Outside support rollers 20 which may be connected to a drive mechanism to improve the process control possibilities are provided at mandrel 15 to sealingly engage the pipe against said mandrel, and a calibrator 21 is provided in the path of the pipe located at a site where the pipe has been expanded. Calibrator 21 forms a passage determining the outside diameter of the finished pipe and provides cooling of the pipe by the supply of cold water which is distributed over the outside surface of the pipe through apertures 22 in the surface of the calibrator which is engaged by the moving pipe. The cooling of the pipe is sufficient to solidify the olefin (co)polymer material so that the pipe when emanating from calibrator 21 is a rigid pipe downstream of the calibrator. A take-up device 23 is provided which engages the outside surface of the rigid pipe and operates to impart to the pipe axial traction. The speed of the take-up device preferably should be adjustable so that the traction force imparted to the moving pipe can be controlled. It should be mentioned that the traction could also in special cases be negative because the pipe gets shorter during expansion if not drawn.

By the hoop stretching of the at least partly cross-linked olefin (co) polymer material by expansion of the tubular member between mandrel 15 and calibrator 21 and the axial stretching of the pipe effected by take-up device 23 the finished pipe should have a relationship between wall thickness and diameter which is  $\geq 3:100$ . The hoop

stretching of the pipe material causes orientation of the olefin (co)polymer material in the hoop direction and this stretching preferably should range from 25 % to 400 % and preferably is 100 %. The axial stretching of the pipe material preferably should range from 0 % to 400 %, and preferably is about 30 %, and causes orientation of the olefin (co)polymer material in the axial direction. By the bidirectional orientation of the olefin (co)polymer material improved strength is imparted to the pipe, and due to the olefin (co)polymer material being at least partly crosslinked when the orientation is effected such orientation can be effected and maintained in a wide temperature range, typically from 135°C to 250°C.

Whereas 10 - 90 % crosslinking degree is enough to increase the orientation temperature range sufficiently further crosslinking of 80 - 0 % can be effected in order to obtain dimensional stability of the pipe in an efficient manner. Such further crosslinking can be effected after expansion of the tubular member on the expanded pipe at a site between calibrator 21 and balloon plug 18 and can be obtained by gamma radiation or electron radiation of the pipe but preferably is obtained by reheating of the extruded pipe at said site provided there is sufficient amount of crosslinking agent left in the polyethylene material after the initial crosslinking obtained by heating of the olefin (co)polymer material in the bipartite molds. Such reheating can be effected by means of circulating trains of heated concatenated mold halves as previously described and subsequent calibration and cooling between calibrator 21 and balloon plug 18. The further crosslinking after orientation of the olefin (co)polymer material provides increased dimensional stability against reversion of the orientation at higher temperature.

Heating of the tubular member immediately downstream of the extrusion head can be dispensed with if the olefin



(co)polymer material is heated sufficiently in the extruder to be kept at the necessary temperature during sufficient time for crosslinking to the desired degree to take place before orientation. It should also be understood that other means for holding the temperature of the extruded tubular member or for reheating the pipe, respectively, than heated circulating mold halves, e.g. a bath or dielectric heating, can be used. However, circulating mold halves are preferred in manufacturing oriented ribbed pipes.

Free radial expansion of the tubular member is applied in the embodiment described but the expansion can also be effected over a mandrel inside a jacket or similar device surrounding the tubular member as shown in FIG 2.

In the embodiment of FIG 2 mandrel 11 is extended to form a mandrel head 11B which widens conically in the flow direction of tubular member 12 to expand said member radially so as to stretch the plastic material in the hoop direction. The conical portion of mandrel head 11B joins a cylindrical portion for inside calibration of the pipe formed by expansion of the tubular member. The mandrel head thus has a substantially S-shaped contour. Die 10 is extended to form a jacket 10A enclosing the tubular member when passing from the extruder to and in over the conical portion of mandrel head 11B. Thus, it will be seen that mandrel head 11B and jacket 10A define a space for the expansion of the tubular member passing therethrough. The surfaces defining said space can be coated with a low-friction material such as polytetrafluorethylene. The jacket can also end close to the point where the conical portion begins. In this case speed controlled rollers could be provided in the vicinity of mandrel head 11B. Jacket 10A can be provided with electric heating elements on the outside thereof for heating the tubular member as may be necessary in order to impart to said member the temperature necessary for the desired crosslinking to take place when

the tubular member is passing through the jacket. Further crosslinking in this case can easily be achieved by extending the heated length of mandrel head 11B.

Furthermore, the end part of jacket 10A can be cooled in order to give a shiny outside to the pipe. Also mandrel head 11B can be heated over the conically widening portion thereof and can be cooled downstream of said portion.

Bar 19 is connected to mandrel head 11B and anchors balloon plug 18 to the extruder said head being located at the entrance end of take-up device 23. As in the embodiment previously described there is in bar 19 passages for supplying a gaseous fluid such as air or inert gas under pressure to balloon plug 18 and to the interior of the pipe formed after expansion of the tubular member. Between mandrel head 11B and balloon plug 18 there are provided nozzles 24 for sprinkling cooling water over the pipe both when it passes over the cylindrical portion of mandrel head 11B and when it has left said portion in order to rigidify the calibrated pipe.

The benefit of the mandrel process described that it can easily be used for both internally calibrated pipes (cooling extension of mandrel head 11B) and for externally calibrated pipes (with similar arrangement as in Fig. 1). The need of plug 18 depends partly also from the lubrication system. In a preferred embodiment the pressurized fluid between plug 18 and mandrel head 11B, which can be used for forcing the still soft pipe against an outside calibrator, can function as a lubricant, at least for the start-up phase, between the inside of the pipe and the outside of the mandrel and the plug, respectively.

The crosslinking process can be initiated already at the end of the extruder, i.e. inside die 10 by any suitable means, e.g. UV, if die 10 is made of glass. Then, the main part of crosslinking is done in the die or the bipartite molds. The exact point where cooling of the pipe starts

after expansion should be chosen with regard to the desired crosslinking in the expanded state.

The processes for orientation of plastic pipes, e.g. the process disclosed in DE 23 57 210, most often include a fairly long conical mandrel. In order to achieve high orientation rates shorter conical parts might be interesting. On the other hand, if the orientation takes place freely i.e. by means of a differential pressure over the wall of the tubular member, then said member will adopt an S-shaped curve, the cross-section being close to parabel shape. This shape, seen so often in film blowing, is a balance of modulus, drawing speed, temperature, wall thickness and draw ratio. Surprisingly, this shape is also effective as mandrel form. Without wishing to be bound by any particular theory, it is believed that a hydraulic lubricating agent, which can be injected to both sides of the tubular member, forms with this shape a natural, well balanced hydrodynamic cushion. The benefit of this form is that the likelihood of the material to drag on the mandrel is reduced. This has been found beneficial in case that no lubricating agents are used, typically at time after start up when the process has stabilized. Then coatings which have good lubricity properties like polytetrafluorethylene, seem to be adequate.

The product which is manufactured by the method of the invention can be a composite product such as a multilayer pipe wherein the layers may be of different plastic materials, or a pipe with axial stripes of different plastic materials. The layers or stripes can be crosslinked or non-crosslinked, and in case they are crosslinked they can include different crosslink agents. The expression "different materials" also includes materials of the same chemical composition but crosslinked to different degrees ranging from 0 to 100 %. In a multilayer pipe having layers of different materials the

layers may have different shrinkage properties, which makes the pipe behave in a very peculiar way when heated, especially if rotating die technology has been used. Some plastic materials such as polyethylene, relax so fast that crosslinking is the almost only way to induce orientation. If a polyethylene pipe in order to have increased strength has an oriented outside layer of crosslinked polyethylene, PEX, and an inside layer of non-crosslinked polyethylene, PE, the composite pipe will bend slightly if heated above the glass transition temperature ( $T_g$ ), depending on e.g. relative wall thickness and centering of the layers. Also, the inside PE layer may prevent the whole pipe from losing diameter when heated. By the addition of fillers such as fibers or flakes (mica) in the PEX and PE layers or in the PE layer the heat deflection temperature (HDT) can be increased. Thus the filled non-crosslinked oriented PE layer can be made strong enough to stand the shrinkage forces originating from PEX layer even at elevated temperature. The incorporation of fillers into the non-crosslinked layer is beneficial at least because the improved thermal conductivity improves the possibilities to prevent too fast relaxation too, hence making permanent orientation easier.

In general fibers very effectively stop the PEX tendency to shrink back, which also makes socketing easier. Hence it can be said that fiber reinforced, crosslinked and oriented pipe must be the top in performance. Naturally the inclusion of fibers into high viscous olefin (co)polymer is not very easy, and therefore a separate layer of softer material, wherein the blending can more readily be done, is highly beneficial. Generally, there are vast possibilities to optimize the strength. Compared to non-oriented homogenous pipes which exhibit the same modulus in all directions, oriented pipes are already an improvement because, for example by varying the draw directions and

ratios, the hoop strength can be easily doubled to that of axial strength - a situation which is commonplace in pressurized pipelines. By adding fillers the possibilities to build up the strength of the composite become multiplied.

A combination of materials wherein an inside non-oriented layer and an oriented PEX outside layer can also be special if the inside layer has a clearly higher melting point than the softening point of PEX, which is around 130°C. This kind of inside material could be special PP grades, which additionally show very sudden softening. This kind of combination could be used as a fast shrink and/or electrofusion sleeve which generates high shrinking power too.

The addition of non-crosslinked material to the both sides of the product can greatly improve the orientation process because these layers can be used to minimize the friction against tooling. When for example silicone oil is mixed only to a thin skin it will not disturb the crosslinking process and the consumption thereof is greatly reduced compared to mixing to the whole bulk of the product. A typical problem in extruding PEX pipes is that residues of peroxide collect in the extrusion head and have to be removed daily. This problem is overcome by having non-crosslinked material on both sides of the product.

In conventional orientation of polyolefins the molecular chains are elongated under influence of the stretching force. On the other hand this phenomenon is counter-balanced by the so-called relaxation, which tends to restore the molecular chains to the coiled, disordered condition. In the process of the invention the cross ties between the chains prevent the extremely rapid relaxation so that the draw speed is not so limited to suitable balance values. However, the material to be oriented will, after crosslinking, at the processing temperature, be at

glassy state and hence rather brittle. Hence the stretching rate must not be too high, because otherwise the melt reacts elastically and because of the brittleness it breaks. It has been found that polyolefin compositions with wide molecular weight distribution do not break so easily. Surprisingly, it has been found that the skin layers on the product, when their material is suitably chosen, greatly enhance the available stretching rates, it seems that the skins can "carry" the brittle layer without ruptures. Similarly the process which does not rely on stretching too much is favoured.

Table I

Degree of crosslinking %	Increase in tensile strength at break %
22	75
33	88
60	116
87	128

The Table I above illustrates the improvement obtained by the method of the invention. The right column indicates the increase in tensile strength at break for PEX samples crosslinked and uniaxially stretched 100 % at 170°C for orientation of the material as compared to non-stretched samples. The table shows the permanent difference in strength of the samples as a parameter of crosslinking degree. It also shows that achieving permanent orientation and enhanced strength properties at high draw temperatures is most unlikely unless the molecules are tied by crosslinking before draw.

The invention facilitates joining of pipes having a spigot end and a socket end, which have been produced by the method of the invention. A sealing ring is mounted to the spigot end of one pipe and is located in the intended position by a metal ring or by double-sided sand paper

wrapped around the pipe. The socket end of the other pipe is widened mechanically, and the spigot end with the sealing ring is pushed into the socket. After a short time, about 15 seconds, the socket has returned to its original  
5 condition clamping the sealing ring between the inside of the socket and the outside of the spigot.

## CLAIMS

1. A method of extruding a product of plastic material, characterized by the steps of

5 adding a chemical crosslinking agent before or during extrusion to the plastic material of the entire product, of one or more layers of a multilayer product, or of axial stripes of the product,

extruding a parison of the plastic material thus prepared,

10 starting crosslinking of the plastic material having crosslinking agent added thereto when the plastic material is in a molten state during extrusion, to a degree ranging from 10 to 100 %,

15 stretching the still soft, at least partly crosslinked parison in one or both of two directions simultaneously or stepwise, said stretching including axial draw to effect orientation of the material in the longitudinal direction of the parison and radial expansion to effect orientation of the material in the hoop direction of the parison, and

20 calibrating and cooling the parison in the stressed condition to make the orientation permanent at least in the crosslinked part.

25 2. The method of claim 1, characterized in that the plastic material to be crosslinked is low density polyethylene.

3. The method of claim 1 or 2, characterized in that the plastic material to be crosslinked is a polyolefin composition comprising an olefin polymer or co-polymer having an average molecular weight (Mw) ranging from 30000 to 1000 000 g/mol and an olefin polymer or co-polymer having a molecular weight greater than 1000 000 g/mol.

35 4. The method of claim 1, characterized in that the plastic material to be crosslinked is high



density polyethylene with a molecular weight (Mw) ranging from 400000 to 15000000 g/mol.

5 The method of claim 4, characterized in that at least the plastic material not to be crosslinked has a wide molecular weight distribution (Mw/Mn) ranging from 5 to 15.

6 The method of any of claims 1 to 5, characterized in that the stretching rates range from 0.002 to 1 s<sup>-1</sup>.

10 7. The method of claim 1 for extruding a polyethylene pipe of improved strength having a relationship between wall thickness and diameter which is  $\geq 3:100$ , wherein the polyethylene material is bidirectionally oriented, characterized in that the parison is extruded as a tubular member.

15 8. The method of claim 7, characterized in that the tubular method is exposed to free radial expansion.

20 9. The method of claim 8, characterized in that the free radial expansion is facilitated by maintaining a differential fluid pressure over the wall of the tubular member.

25 10. The method of claim 7, characterized in that the tubular member is exposed to radial expansion on a mandrel.

11. The method of claim 10, characterized in that the tubular member is enclosed by a jacket either heated or cooled, during expansion.

30 12. The method of any of claims 7 to 11, characterized in that stretching of the polyethylene material of the tubular member in the hoop direction ranges from 25 % to 400 %.

35 13. The method of claim 12, characterized in that stretching of the polyethylene material is about 100 %.

1995-09-20

Huvudfaxen Kassar

14. The method of any of claims 7 to 13, characterized in that stretching of the polyethylene material of the tubular member in the axial direction ranges from 0 to 400 %.

5 15. The method of claim 14, characterized in that stretching of the polyethylene material of the tubular member in the axial direction is about 30 %.

10 16. The method of claim 1, characterized in that the tubular member during crosslinking is kept against molds moving with said member by differential fluid pressure over the wall of said member.

15 17. The method of claim 16, characterized in that said molds are heated for holding the temperature of the polyethylene material at crosslinking temperature.

18. The method of any of claim 7 to 17, characterized in that the polyethylene material after said initial partial crosslinking is further crosslinked after orientation of the polyethylene material.

20 19. The method of any of claim 7 to 18, characterized in that the polyethylene material is HD polyethylene with the addition of peroxides and phenolic antioxidants amounting to 0.1-0.7 % by weight, preferably 0.25-0.5 %.

25 20. The method of any of claims 1 to 19, characterized in that the extent to which the product is crosslinked and oriented is controlled by choosing the starting point of cooling in relation to the point where the final dimension of the product is reached.

30 21. The method of any of claims 1 to 20, characterized in that an inside surface of the product is substantially cooled to impart to said surface high gloss and smoothness.

35 22. A hollow product of plastic material forming one or more layers, characterized in that at least

part of the product is both crosslinked and permanently oriented at ambient temperature.

23. The product of claim 22,  
c h a r a c t e r i z e d in that said part forms one or  
5 more stripes along the axis of the product.

24. The product of claim 22,  
c h a r a c t e r i z e d in that said part forms  
concentric layers around the axis of the product.

25. The product of claim 24,  
10 c h a r a c t e r i z e d in that an inner layer of the  
product is a non-crosslinked layer impermeable to rest  
products from crosslinking of other layers of the product.

26. The product of claim 24 or 25,  
c h a r a c t e r i z e d in that the product has an  
15 outside skin of plastic material which is substantially  
non-oriented, the thickness of said skin being 0.001 to 3  
mm.

27. The product of claim 24 or 26,  
c h a r a c t e r i z e d in that the product has an  
20 inside skin of plastic material which is substantially non-  
oriented, said skin having a thickness of 0.01 to 10 mm.

28. The product according to any of claims 22 to 27,  
c h a r a c t e r i z e d in that the non-oriented part or  
parts and the oriented part or parts are made of the same  
25 polymer.

29. The product of any of claims 22 to 27,  
c h a r a c t e r i z e d in that the non-oriented part or  
parts consist of a polymer that is different from that of  
the oriented part or parts.

30. The product of claim 22,  
30 c h a r a c t e r i z e d in that the product forms a pipe  
having a relationship between wall thickness and average  
diameter which is  $\geq 3:100$ .

31. The product of any of claims 22 to 30,  
35 c h a r a c t e r i z e d in that the oriented and

1995-09-20

Huvudfaxen Kassar

crosslinked part or parts make up more than half the product volume.

32. The product according to claim 22,  
c h a r a c t e r i z e d in that the oriented and  
5 crosslinked parts are located on both sides of one or more  
non-oriented layers, said non-oriented layer or layers  
having barrier properties different from those of the  
oriented and crosslinked layer or layers.

33. The product of claim 22,  
10 c h a r a c t e r i z e d in that the plastic material of  
the oriented and crosslinked part or parts consists of a  
polyolefin composition comprising an olefin polymer or  
(co)polymer having an average molecular weight (Mw) ranging  
from 30000 to 1000 000, and an olefin polymer or  
15 (co)polymer having a molecular weight greater than  
1:000 000 g/mol.

34. The product of any of claims 22 to 33,  
c h a r a c t e r i z e d in that one or more parts of the  
product contain fillers as fibers or flakes, which are also  
20 oriented.

35. The product of claim 22,  
c h a r a c t e r i z e d in that it comprises an oriented,  
crosslinked polyethylene pipe with hydrostatic design base  
of at least 16 Mpa.

25 36. An extruder line for the manufacture of products  
with one or more layers with crosslinked and oriented  
sections of plastic material, c h a r a c t e r i z e d in  
that the extruder comprises a mandrel which is fixed in  
such a manner that the cross-section of the flow passage is  
30 kept substantially constant from the screw end to the  
point, where stretching of the plastic material starts.

37. The extruder line of claim 36,  
c h a r a c t e r i z e d in that the mandrel is supported  
by the body of the extruder via the screw.

38. The extruder line according to claims 36 or 37,  
c h a r a c t e r i z e d in that the flow passage is free  
of obstacles.

39. The extruder line of any of claims 36 to 38,  
5 c h a r a c t e r i z e d in that the diameter of the  
mandrel is substantially constant from the extruder outlet  
to the point where stretching starts.

40. The extruder line of any of claims 36 to 39,  
c h a r a c t e r i z e d in that that the mandrel forms a  
10 conically widening portion.

41. The extruder line of claim 40  
c h a r a c t e r i z e d in that a jacket extends partly  
over said conically widening portion.

42. The extruder line of claim 41,  
15 c h a r a c t e r i z e d in that said conically widening  
portion and said jacket are coated with  
polytetrafluorethylene.

43. The extruder line of claim 40  
c h a r a c t e r i z e d in that the conical widening  
20 portion of the mandrel joins a cylindrical portion of the  
mandrel along an s-shaped contour.

44. The extruder line of any of claims 40 to 43,  
c h a r a c t e r i z e d in that the mandrel is heated  
over a portion, including said conically widening portion,  
25 and downstream thereof is cooled.

45. The extruder line of any of claims 36 to 44,  
c h a r a c t e r i z e d in that to the end of the  
mandrel there is connected a shaft (19) which comprises  
fluid inlets and outlets, and a gasket (18) the diameter of  
30 which can be adjusted from the outside of the extruder  
line.

46. A method of joining two pipes according to any of  
claims 22 to 34, said pipes each have a spigot end and a  
socket end, c h a r a c t e r i z e d in that a sealing  
35 ring is mounted to the spigot end of the pipe, that the

socket end of the other pipe is widened mechanically, and that said spigot end is inserted in the widened socket end, said socket end returning to the original condition thereof clamping the sealing ring between the outside of the spigot  
5 end and the inside of the socket end.

47. The method of claim 46, c h a r a c t e r i z e d in that means is provided between the spigot end and the sealing ring to locate the sealing ring axially on the spigot end.

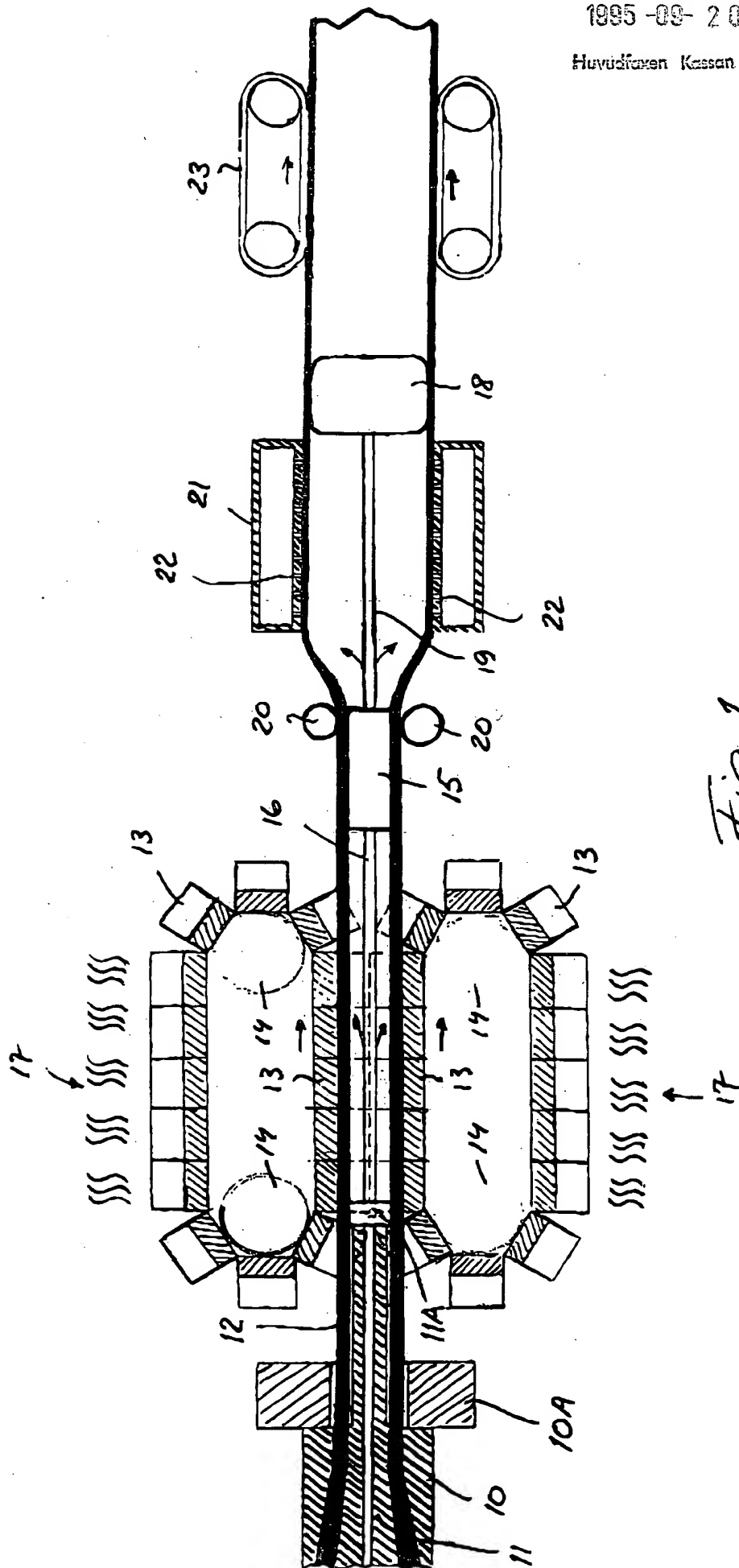
## ABSTRACT

A method of extruding a polyethylene pipe of improved strength having a relationship between wall thickness and diameter which is  $\geq 3:100$ . A chemical crosslinking agent is added to the polyethylene material to be extruded, and the polyethylene material is crosslinked in the molten state thereof to a degree ranging from 10 to 100 % during extrusion of a tubular member. The soft polyethylene material at least partly crosslinked is stretched in the hoop direction of the tubular member by radial expansion thereof to effect orientation of the polyethylene material in the hoop direction, and in the axial direction of the tubular member to effect orientation of the polyethylene material in the axial direction. Then, the pipe thus obtained is calibrated and cooled.

Ink. t. Patent- och reg.verket

1995-09-20

Huvudföres. Kassen

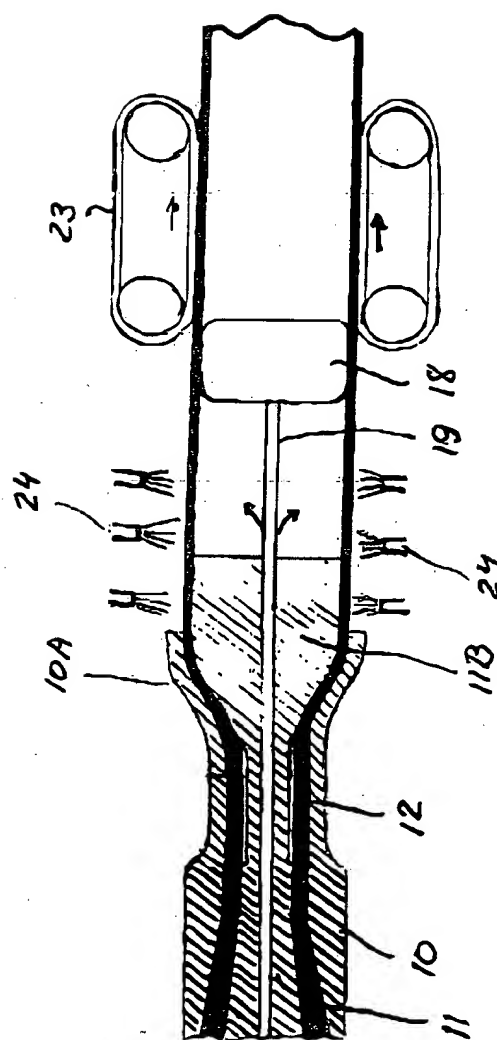




Ink. t. Patent- och reg.verket

1995-09-20

Huvudfören. Kassa



**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**